National Bureau of Standards

TECHNICAL NEWS BULLETIN

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Constant-Level Device for Liquids

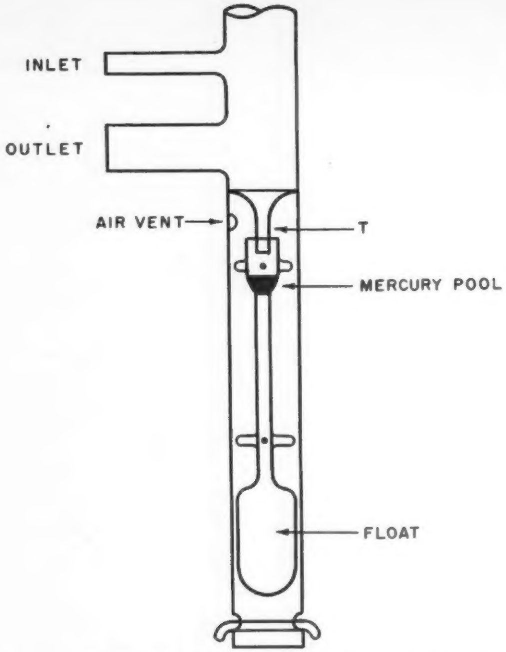
A COMPACT, easily constructed device for maintaining a constant liquid level in laboratory apparatus has been recently developed by D. E. Couch and Abner Brenner of the National Bureau of Standards. Although designed primarily for small installations, the leveler should also prove useful on large-scale equipment.

While several devices for maintaining a constant level of liquid in tanks are commercially available, none of these are satisfactory for use with small laboratory set-ups, such as electroplating baths operated at elevated temperatures Usually they employ a siphon arrangement or make use of an electrical circuit to operate a solenoidal valve or pump. Those siphon levelers which receive water directly from the tap are not adaptable to plating baths, because they siphon off part of the bath when the level is raised by the addition of a large piece of work. The electrical devices, besides being rather expensive, are not sufficiently portable for use with small baths. The Bureau's leveler eliminates many of the disadvantages of the previous types. It takes water directly from the tap, is of inexpensive construction, and can be easily moved from one installation to another.

The NBS leveler employs a water inlet and an overflow, or outlet pipe, to maintain a constant head. A mercury valve, attached to a float, allows the water to flow into the bath if the bath level is lowered and prevents flow outward when the level of the bath rises.

The device takes the form of a vertical tube with inlet and outlet connections near the top. Just below the outlet connection the tube is sealed off except for a smaller-diameter open tube which extends below this point, connecting the upper section of the larger tube with the lower. An open cup of mercury is supported below the small connecting tube by a float which rises and falls with the liquid level in the lower section of the larger tube. When the bath level rises, the float also rises, causing the mercury cup to seal off the lower end of the small connecting tube. All of the water entering through the inlet is then rejected and is eliminated through the outlet connections. When the bath level drops, the float descends, taking with it the mercury cup, and the water runs into the bath through the small tube.

Levelers of this kind can be made in any size, for solution depths of 10 cm or more. None of the dimensions are critical; however, the tube should be of sufficient size that it will not become clogged by the small particles of sediment that may enter with the tap water, and the mercury cup must be large enough and so centered that it will slide freely up over the small tube without touching. A convenient diameter for the larger tube is 25 mm. In order for the water to flow freely, the overflow tube should be about 2 cm in diameter and the float should have about 3 mm clearance with the surrounding tube.



Schematic diagram of the device developed by the Bureau for maintaining a constant liquid level in electroplating baths. Water from the tap enters through the inlet tube and flows out through the outlet tube just below. When the bath level drops, the float goes down, and water from the tap runs into the bath through the small tube *T*. Then, as the bath level rises, the mercury pool comes up to close off the tube *T*, and all the tap water passes out through the outlet. A small air vent prevents the column from flooding with water.

At NBS, one of the new levelers has been used on a 6-liter bath operating at 85° C, and another has been employed with a chromium-plating bath of commercial size. Both have functioned satisfactorily for several months, maintaining liquid levels constant to within 5 mm.

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Charles Sawyer, *Secretary*

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A. V. Astin, *Director*

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Contents

Fluorescence of Teeth Reveals Structure

A NEW TECHNIQUE that utilizes the natural fluorescence of human teeth is being employed at the Bureau to reveal hitherto undetected details of tooth structure. In the NBS method, fluorescent photomicrographs are obtained from the visible light emitted by very thin tooth sections under high-intensity ultraviolet radiation. The micrographs provide information on the distribution of the organic content of enamel and dentin and show developmental or growth lines very clearly. Such information is expected to be of value in explaining the mechanism of decay.

Recording spectrometer used to analyze the fluorescence of tooth structures. Ultraviolet radiation from the mercury-vapor lamp (upper left) excites fluorescence in a tooth section mounted in the microscope (right of the lamp). The microscope projects an image of the fluorescent tooth section onto the slit of the spectrometer, and the spectral intensities of various areas of the specimen are automatically recorded on the chart at right.

Great differences exist in the resistance of the teeth of different individuals to decay, and these differences are thought to be associated in some way with the manner in which the hard, calcified tooth structures are deposited. To learn more about the problem, the

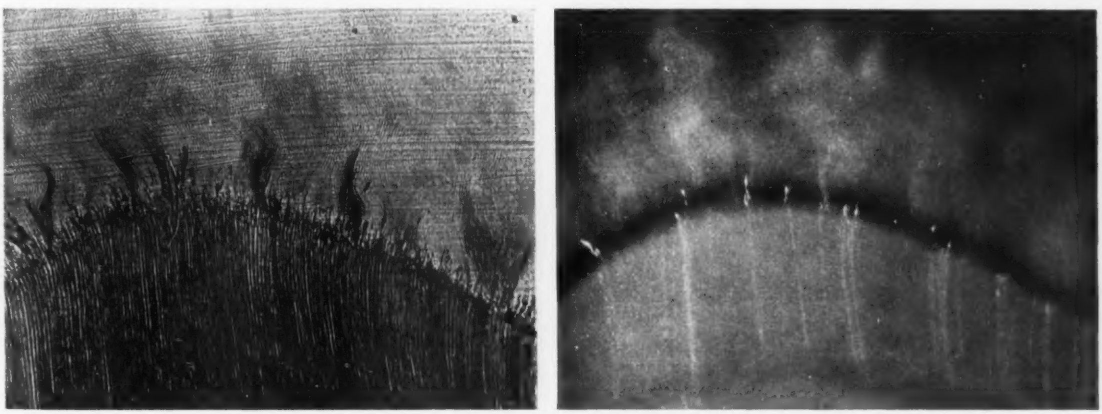
Fluorescent photomicrograph of a tooth section irradiated with ultraviolet light. Note that the dentin (inner portion of the tooth) fluoresces with much greater intensity than the outer enamel and that lines of growth in the dentin stand out with great clarity.

Bureau, in cooperation with the American Dental Association, has been investigating the structure of teeth by a variety of physical and chemical techniques. For many years it has been known that human teeth fluoresce, emitting visible light, when irradiated with ultraviolet light. Since in general the fluorescence of a substance is very sensitive to small difference in structure or composition, it was felt that a thorough investigation of the fluorescence of enamel and dentin might bring out details of structure not made apparent by other methods. The Bureau therefore began the development of methods and equipment for exciting and analyzing the fluorescence of tooth structures. The fluorescence technique was developed by George Dickson and I. C. Schoonover of the NBS staff and A. F. Forziati of the American Dental Association Research Fellowship at the Bureau.

Preliminary investigations showed that the fluorescence of teeth is excited strongly by radiation in the region of 3,650 A. Attempts to photograph the emitted light and to examine it spectrographically soon showed the need for a high-intensity source of ultraviolet radiation to produce sufficient intensity in the fluorescence. Ultimately a 1,000-watt water-cooled mercury-vapor lamp with a quartz jacket was selected. This lamp operates at 1,000 volts and at a pressure of approximately 2,000 lb/in.[2] Glass filters that transmit radiation in the region between 3,200 and 3,900 A are used to absorb the visible light from the lamp, and a quartz condensing lens and a front-surface mirror concentrate the ultraviolet radiation on the specimen.

In preparing the specimens, serial sections approximately 200 microns thick are first cut by means of a high-speed water-washed carborundum disk, 0.015 inch thick. These sections are then ground under water to a thickness of 25 to 75 microns on a polishing instrument, which consists of a diamond-impregnated disk rotating on an axis perpendicular to the plane of a reciprocating platform. Suction, operating through small holes in the platform, holds the specimens in place during the grinding. The thin sections are cemented to microscope slides, using sodium silicate as a mounting medium since it does not fluoresce as do most of the common mounting materials.

Fluorophotomicrographs, showing variations in intensity of the fluorescence of different areas of teeth, are made with a photomicrographic camera used with either camera lenses or a microscope, depending upon



Dentin-enamel junction in a tooth section, photographed by visible light (left) and by a new NBS technique (right) which utilizes the fluorescence excited in the tooth by ultraviolet radiation. Note the absence of fluorescence in the immediate area of the junction and the intense fluorescence of the tufts extending upward into the enamel.

the magnification desired. Glass filters, carefully selected for suitable absorption and transmission bands and for absence of fluorescence, are placed between the specimen and the film to prevent scattered ultraviolet radiation from fogging the film. These photomicrographs show clearly details of tooth structure not made evident by other methods of investigation.

The spectra of the fluorescence of tooth structures have also been investigated. A recording spectrometer is used in which a photomultiplier tube measures the radiant energy in the various parts of the spectrum. For studies of the fluorescence of large areas, the specimen, either a thin section or a fine powder cemented to a microscope slide, is mounted directly in front of the spectrometer slit and irradiated with ultraviolet light. To investigate the fluorescence of small areas, a microscope is used to project an image of the area under study onto the slit of the spectrometer.

Results obtained with large specimens indicate that dentin fluoresces approximately four times as intensely as enamel and that the fluorescence of enamel contains more of the yellow wavelengths than does that of dentin. Studies of smaller areas have revealed a number of regions having a noticeably higher fluorescence than the rest of the tooth structure. The greatest intensity has been observed near the enamel-dentin junction, along lamellae, and to a shallow depth below the exposed surfaces of the enamel. Efforts are now being made to obtain more detailed information on the fluorescence spectra of small areas.

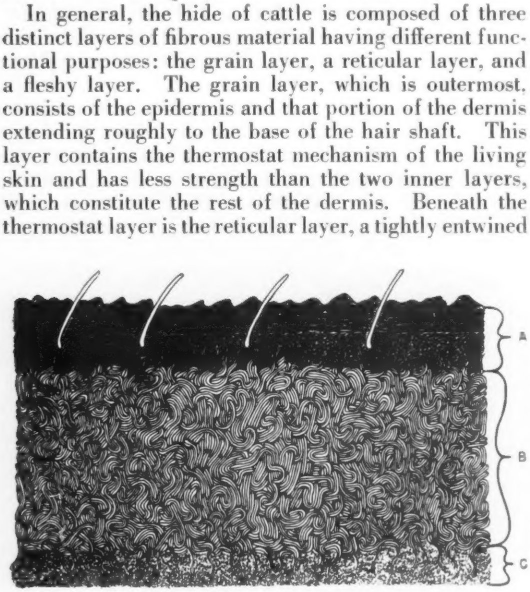
Strength of Split Leather

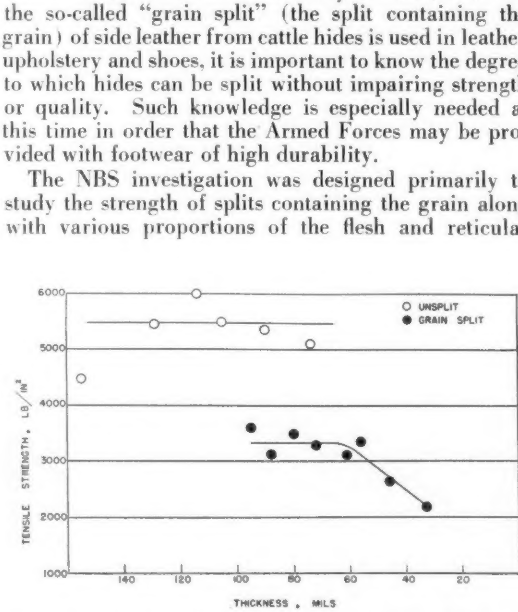
A MUCH clearer picture of the effect of splitting on the strength of leather has resulted from a recent study conducted by Dr. J. R. Kanagy and associates at the Bureau. From a statistical treatment of the experimental data, a simple formula has been derived which gives the breaking strength of a split specimen of chrome-tanned cowhide in terms of its thickness. The investigation was carried out as part of a broad research program on the physical properties and sampling of shoe upper leather, sponsored at NBS by the Office of the Quartermaster General.

In general, the hide of cattle is composed of three distinct layers of fibrous material having different functional purposes: the grain layer, a reticular layer, and a fleshy layer. The grain layer, which is outermost, consists of the epidermis and that portion of the dermis extending roughly to the base of the hair shaft. This layer contains the thermostat mechanism of the living skin and has less strength than the two inner layers, which constitute the rest of the dermis. Beneath the thermostat layer is the reticular layer, a tightly entwined mass of large, strong fibers. The fibers of the reticular layer untwine and become smaller as they enter the thermostat layer and then finally fade out to form a more or less plastic mass at the surface of the grain. The reticular fibers also untwine and become smaller as they enter the fleshy layer directly beneath the reticular layer. The reticular layer is thus the strongest of the three layers.

When leather is made thinner by splitting, the relative proportions of the layers change, and the leather becomes weaker as the reticular layer is reduced. As the so-called "grain split" (the split containing the grain) of side leather from cattle hides is used in leather upholstery and shoes, it is important to know the degree to which hides can be split without impairing strength or quality. Such knowledge is especially needed at this time in order that the Armed Forces may be provided with footwear of high durability.

The NBS investigation was designed primarily to study the strength of splits containing the grain along with various proportions of the flesh and reticular





Left: **drawing showing the characteristic layers in a cross section of cowhide. *A:* grain layer, consisting of the epidermis and a small portion of the dermis. *B:* reticular layer, containing large intertwined fibers. Most of the strength of the leather is due to this layer. *C:* fleshy layer. *Right:* tensile strength of unsplit (open circles) and grain-split (closed circles) leather plotted against thickness. The abrupt drop in tensile strength caused by splitting is probably due to the cutting of the ends of the reticular fibers, which are anchored to some extent in the fleshy layer of the hide.**

layers. However, tests were also made on split-off specimens of different thicknesses containing only the flesh and a portion of the reticular layer.

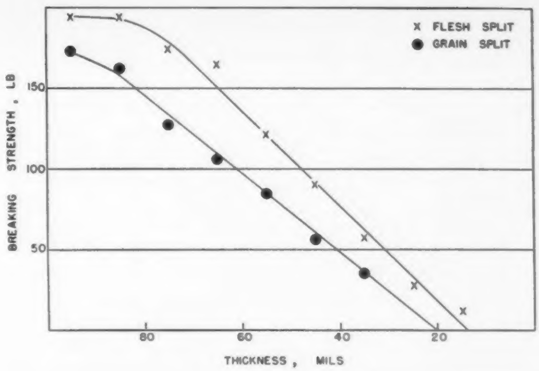
Ten commercial chrome-tanned steer sides were cut into 7 x 7-inch blocks. Specimens from each side were split to three different thicknesses while other specimens from the same side were left unsplit to serve as controls. All were subjected to tests of tensile and breaking strengths. In this way, results were obtained for the effect of thickness on strength over the entire area of the side.

From the data it was evident that unsplit leather is much stronger than split leather of the same thickness and that the strength of the split leather increases with thickness. When either breaking strength or tensile strength of the split leather specimens was plotted against thickness, a straight line was obtained which could be extrapolated to give an intercept on the thickness axis. This intercept was assumed to be due to the effect of the grain layer, which has very low strength. It was also found that for equal thicknesses split leather containing the bottom flesh layer is stronger than that containing the top grain layer.

Splitting was accompanied by an abrupt drop in tensile strength, probably as a result of the cutting of the ends of the reticular fibers, which appear to be anchored to some extent in the flesh layer. The cut fibers might be expected to pull apart more readily causing instability in the hide structure and a corresponding reduction in tensile and breaking strength.

To obtain more nearly correct values for the slope and intercept of the curve of breaking strength versus thickness for the grain split, least squares procedures were applied to the average results for each location in a given side. The results for each location were then averaged to give an average for the entire side. In this way, the following equation relating strength and thickness of split grain leather was obtained:

$$\text{breaking strength (in lb)} = 3 \times \text{thickness (in mils)} - 100.$$



Comparative strengths of split cowhide containing the bottom flesh layer (X's) or the top grain layer (closed circles). For equal thicknesses, the fleshy layer is obviously the stronger. On extrapolation, the leathers having the grain layer intact show an intercept at a thickness of about 20 mils while those having the flesh layer intact have an intercept at about 15 mils. The latter intercept is assumed to represent the minimum thickness of a sheet of leather split on both sides which would have sufficient fiber strength to hold together under negligible load.

From these results, the average value for the intercept was found to be 32.8 mils. By the use of this simple formula, it can be easily shown, for example, that a split specimen of shoe upper leather or upholstery leather of the type studied having a thickness less than 60 mils would have a strength of less than 80 lb.

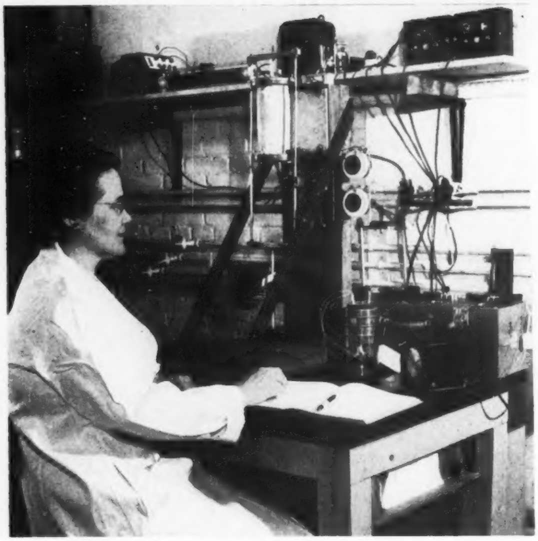
For further details, see Influence of splitting on the strength of chrome-tanned steer hides, *by J. R. Kanagy, W. H. Leser, E. B. Randall, T. J. Carter, and C. W. Mann, J. Am. Leather Chem. Assoc.* **47**, 329 (1952).

Rheology of Concentrated Rubber Solutions

RECENT NBS investigations of the rheology of moderately concentrated rubber solutions using the McKee consistometer have provided flow data on these substances at higher shearing stresses and rates of shear than heretofore possible with conventional viscometers. Directed by A. B. Bestul of the NBS engines and lubrication laboratory and under sponsorship of RFC's Synthetic Rubber Division, this work and companion measurements with several conventional Bingham capillary viscometers have yielded significant information about the fundamentals of the flow behavior of polymer solutions, which are well known to be non-Newtonian fluids—that is, to exhibit a nonlinear relationship between shearing stress and rate of shear.

The development and extensive commercial use of the various types of synthetic rubber have made it necessary to learn more about their flow properties both from the standpoint of industrial processing of the raw rubber and of over-all quality in the finished products. In the past, chiefly because of the lack of suitable instruments, it has been possible to measure the flow properties of bulk rubber only to a very limited extent. High rates of flow could be measured only on relatively dilute solutions; otherwise the material had to be subjected to slow deformation. Through the use of the McKee worker-consistometer, originally developed at the Bureau to study the flow characteristics of greases, measurements can be made for the first time of the viscosities of solutions containing 3 to 100 percent of rubber at widely different rates of flow.

Poiseuille's well-known treatment of capillary flow arrives at the viscosity of a Newtonian fluid by calculating the shearing stress and the rate of shear at the



The McKee worker-consistometer used at NBS to investigate the rheology of rubber solutions. Originally developed at the Bureau to study the effect of mechanical working on greases, the apparatus has greatly extended the range over which viscosity measurements can be made on moderately concentrated polymer solutions.

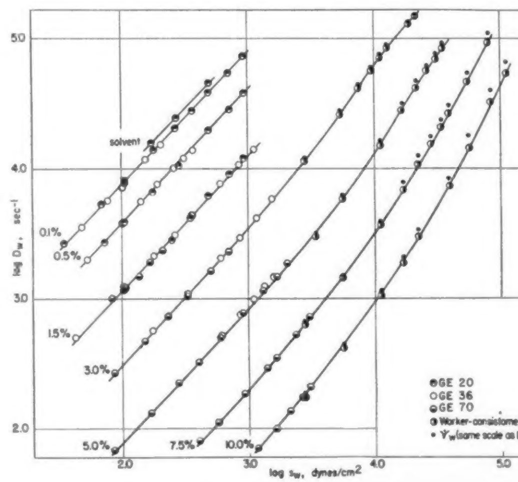
capillary wall and taking the ratio of these two quantities. The expression which gives the shearing stress at the capillary wall in Newtonian capillary flow also gives the shearing stress at the wall in non-Newtonian capillary flow. However, the expression which gives the rate of shear at the capillary wall in Newtonian capillary flow does not give the actual rate of shear at the wall in non-Newtonian capillary flow. Even so, in non-Newtonian flow the quantity obtained from this expression is still a useful quantity and is usually referred to as the nominal rate of shear. It plays the role of a correlating function, because, if the same liquid is investigated in several capillaries of different dimensions, a given value of the shearing stress at the wall must always coexist with the same value of the nominal rate of shear.

When solutions of 3 to 10 percent GR-S rubber are examined with the two types of viscometers, plots of nominal rate of shear versus shearing stress at the wall coincide. Thus the validity of the various corrections applied to the consistometer data (such as that for the resistance of the pistons to sliding in the cylinders) is assured in this region, which lies in the lower part of the ranges of viscosity and of shearing stress.

In Newtonian flow the general relation existing between rate of shear and shearing stress is one of simple proportionality; the ratio of rate of shear to shearing stress, which may be called the fluidity, is a constant. In attempting to express the corresponding (nonlinear) relation existing in non-Newtonian flow a common procedure is to add to the constant further terms of a power series in shearing stress, of which the constant is the zero-order term. All quantitative theories of non-Newtonian flow in polymer solutions indicate that in the limit of zero shearing stress the nonlinear relation must reduce to the linear one of Newtonian flow. This requires that the derivative of fluidity with respect to shearing stress become zero at zero shearing stress. This condition can exist only if there is no first-order term in the extended power series, and the simplest compatible relation is one which adds only the second-power term in shearing stress.

Combination of data from the McKee consistometer and from Bingham viscometers results in flow data for 0.1 to 10 percent solutions of GR-S rubber in *o*-dichlorobenzene over an unusually large range of shearing stress and rate of shear. These data cannot be represented by the relation which adds only the second-power term in shearing stress, but they do fit the corresponding equation which adds only the first-power term. Other, less extensive, flow data in the literature also agree with the first-power relation but not with the second-power relation.

This situation constitutes a serious discrepancy between present theory and experiment. Because the experimental data supporting the first-power relation have been obtained with several different types of viscometers, they are probably valid. If the theories underlying the second-power relation are valid, a possible resolution of the discrepancy may be as follows: the condition of zero derivative of fluidity with respect to shearing stress at zero shearing stress is approached only at shearing stresses much lower than those examined experimentally to date. If this were the case, the second-power relation would fit experi-



Graph showing logarithmic plots of nominal rate of shear at the capillary wall (D_w) versus shearing stress at the wall (s_w) for data obtained on eight different GR–S solutions using three different Bingham viscometers and the McKee consistometer. The coincidence of data for a given solution with viscometers of different capillary dimensions demonstrates that the use of the above flow parameters results in good correlation of such data for both non-Newtonian and Newtonian materials.

mental data taken at shearing stresses much lower than those already examined, although it does not fit present data. In this case the first-power relation should not be compared with theory since the region of validity would be known to be beyond the range of applicability of the theory. If on the other hand the first-power relation is valid at infinitesimal shearing stresses, the concepts of the theory underlying the second-power relation will have to be modified. The questions posed here can be answered only by further flow measurements at shearing stresses lower than have been made to date. Further work in progress at the National Bureau of Standards, primarily on bulk rubbers and high-concentration solutions, is expected to throw additional light on the rheology of polymers and their solutions.

For further technical details see Flow behavior of concentrated GR–S rubber solutions, *by A. B. Bestul and H. V. Belcher, J. of Colloid Science, Vol.* **5**, *No. 3, page 303 (June 1950).*

For complete description of the McKee consistometer see ASTM Bull. No. **153**, *90 (1948) and National Bureau of Standards Tech. News Bull.* **33**, *10 (1949).*

Crystal-Growing Technique

AN IMPROVED technique for growing small crystals of high-melting compounds is being used at the National Bureau of Standards. In the new method, a hot-thermocouple apparatus simultaneously heats the specimen and measures its temperature, while the operator watches crystal growth through a stereoscopic microscope and manually controls the rate of cooling. The new method was devised by Fred Ordway of the Portland Cement Association Fellowship at the Bureau to meet needs arising in an X-ray diffraction study of portland-cement clinker. However, it should also be applicable to crystallographic studies in other fields.

An X-ray diffraction study requires a suitable single-crystal specimen. The ideal specimen should be pure, free from flaws or twinning, and sufficiently well developed that the crystallographic axes can be found from the orientation of the edges and faces. Because large size is not necessary—crystals only 0.05 to 0.1 mm in least dimension can be used with ordinary X-ray cameras—usable crystals of many substances can be found in natural minerals or in products of manufacture. Occasionally, however, all available specimens of a mineral are either too impure or too finely crystallized, and synthetic melts of the proper composition likewise form crystals that are too small. This is true of the calcium silicates and aluminates of portland-cement clinker being studied in the NBS laboratory, and it is this problem that the new technique was devised to solve.

Earlier work had indicated that single crystals are grown most satisfactorily by manually controlling the cooling of the smallest droplet that will yield a crystal large enough to be used. With a droplet of small size, the possibility of growth of unwanted crystals is minimized. Furthermore, the droplet can be observed through a stereoscopic microscope during the entire crystallization process, which may take several hours. Twins or other extraneous crystals can thus be detected and melted away as soon as they form.

The NBS technique facilitates precise control of the cooling process. A thermocouple of platinum and platinum-10%-rhodium is mounted under a stereoscopic microscope. The thermocouple is heated with 5-kilocycle alternating current, supplied through a blocking capacitor. The capacitor prevents the a-c power supply from shorting the direct-current output of the thermocouple, which is measured on an external microammeter that can be calibrated in degrees Centigrade. A small amount of the material under study is placed on the thermocouple, melted, and carefully cooled while crystal formation is watched through the microscope. Although precise control of the cooling process does not require knowledge of exact temperatures, calibration of the NBS apparatus for melting-point determinations indicates a maximum measurement error of only 5° C. in the range 770° to 1,420° C.

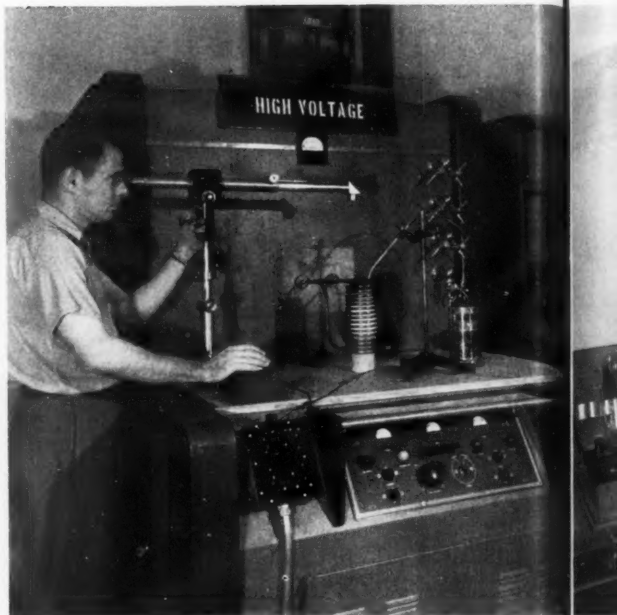
NOTE.—*The crystal-growing apparatus is described in more detail in* Techniques for growing and mounting small single crystals of refractory compounds, *by Fred Ordway, J. Research NBS* **48**, *152 (February 1952), RP2299.*

Small single crystals of high-melting compounds are grown by an improved technique developed at NBS. A small amount of the compound being studied is placed on a thermocouple and melted by heating the thermocouple with alternating current. The direct-current output of the thermocouple simultaneously gives a temperature indication. While watching crystal growth through a binocular microscope, the operator controls the thermocouple temperature, and thus the cooling rate, by means of a potentiometer (right).

Standards Te

AS A RESULT of the increasing need for reliable temperature measurements at both very high and very low temperatures, the National Bureau of Standards is extending its temperature standardization program toward the extremes of the temperature scale. New instruments and methods of calibration are being developed for these regions, and research efforts are being directed toward the extension of the International Temperature Scale to provide greater accuracy and reproducibility in the measurements thus made possible. This work is part of a continuing NBS program, now under the direction of Dr. R. E. Wilson of the Bureau's temperature measurements section, which seeks to provide American science and industry with temperature standards and measurement methods over as broad a range and to as high precision as possible.

While the accurate measurement of temperature has long been of interest to the research scientist, its importance, particularly at the extreme ends of the scale, has greatly increased with the rapid technical developments of recent years. During this period, many improvements in processes for the production of steel, glass, gasoline, and other important commodities have resulted from the increased precision that is being attained in the measurement and control of temperature. In aeronautics there are new temperature problems in connection with the use of jet propulsion and the operation of aircraft at high altitudes and in polar regions. To attain the desired performance in jet engines, fundamental research is required to develop methods of measuring temperatures of flames and to develop temperature-sensitive devices for indicating performance and controlling operation. For use at



Left: **experimental high-temperature calibration of a ther ple in being used to determine the temperature of the thermocou junc radio-frequency heating coil (center). The cold junction in an** ***Right:*** **optical pyrometers are calibrated by comparison wit dard filament lamp (background), which serves as a constant-te e sou**

jet-engine temperatures as well as in arctic cold, mechanical parts must be specially designed using materials capable of withstanding these extremes in order to function satisfactorily. Likewise, as a result of the applications of atomic energy, it has become necessary to learn more about the heat-transfer properties of a variety of materials at increasingly high temperatures. For effective research in these fields, temperatures must be accurately measured, and the results of one laboratory must be comparable with those of another.

To provide a fundamental basis for precise temperature measurements, a scale of temperature has been established which covers the range from the boiling point of oxygen to the highest temperatures of incandescent bodies and flames. This scale, known as the International Temperature Scale, is based on six reproducible equilibrium temperatures, or "fixed points," to which numerical values are assigned, and upon specified interpolation formulas relating temperature between or above these points to the indications of standard temperature measuring instruments.

The International Temperature Scale was first adopted in 1927 to provide a scale which would con-

Before testing, liquid-in-glass laboratory thermometers are first examined for dirty mercury and foreign material in the bulb and capillary.

ds Temperature

f a the ple in the NBS pyrometry laboratory. An optical pyrometer is ermocou junction, which is contained in a graphite crucible within a nction i in an ice bath within the Dewar to the right of the heating coil. son wit dard pyrometer. Both instruments are sighted on a ribbon-tant-tem e source.

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form as closely as possible to a thermodynamic scale proposed many years earlier by Lord Kelvin. The scale which he proposed was based on a relation between the efficiency of a reversible heat engine and the temperatures between which the engine operates. This scale is independent of the working substance in the engine. Temperatures on this scale are identical with those appearing in the ideal gas law and other thermodynamic relations. By observing the change of pressure with temperature of a constant volume of gas, or the change in volume of a quantity of gas under constant pressure, and applying a correction for the imperfection of the gas, temperatures on the thermodynamic (Kelvin) scale can be determined. In practice, however, the complexity of an accurate gas thermometer and the difficulty of making highly precise measurements with it are severe limitations on its use.

The experimental difficulties involved in measuring temperature on the thermodynamic scale and the importance of measuring temperature with high precision on the same scale in all countries ultimately led to the establishment of the International Temperature Scale of 1927. This scale, proposed by the national laboratories of the United States, Great Britain, and Germany, and adopted by 31 nations, was designed to conform as nearly as practicable to the thermodynamic scale as it was then known. It has always been possible to obtain values of temperature on the International Temperature Scale more accurately than on the thermodynamic scale.

The International Temperature Scale of 1927 proved useful in providing a stable, uniform, and precise basis for obtaining temperatures. However, in the 20 years following its adoption, the increasing precision attained in temperature measurements made it apparent that some revision was desirable in order to make the scale more self-consistent and to improve its agreement with the thermodynamic scale. The major responsibility for proposing and obtaining agreement on the changes fell to the National Bureau of Standards. After many consultations with scientists and laboratories in this country and abroad, the Bureau prepared a draft that formed the basis of the document finally adopted at Paris by the Ninth General Conference on Weights and Measures as "The International Temperature Scale of 1948" [1, 2]. On January 1, 1949, the Bureau began using the definitions of the 1948 scale, both in its own research program and in calibrating instruments for other scientific and industrial purposes.

The six fixed points of the 1927 scale were the boiling point of oxygen (−182.97° C), the freezing and boiling points of water, the boiling point of sulfur (+444.60° C), the melting point of silver (+960.5° C), and the melting point of (+1,063° C). From −190°

Determination of the ice point of a liquid-in-glass thermometer at NBS. The ice point serves as a reference for determining changes in the volume of the bulb with time.

Liquid-in-glass thermometers are shown being tested by comparison with one of the Bureau's standard platinum resistance thermometers. For the temperature range from 0° to 100° C, the thermometers are immersed in a mechanically stirred water bath. The power input to the heater is controlled by the variable transformer at the left of the bath. The thermometers under test are read with a telescope, and the corresponding reading for the platinum resistance thermometer is obtained with a Mueller thermometer bridge.

to +660° C, the measurement of temperature was based on the indications of a standard platinum resistance thermometer used in accordance with specified formulas; from +660° C to the gold point, the platinum versus platinum-10%-rhodium thermocouple was standard; and above 1,063° C, the optical pyrometer was used.

The same fixed points, with one slight modification, are specified in the 1948 scale, and the instruments and interpolation equations for obtaining temperatures between fixed points are essentially the same as those previously used. Only two revisions in the definition of the scale resulted in appreciable changes in the numerical values assigned to measured temperatures. One of these was the change in the value for the silver point from 960.5° to 960.8° C, which made numerical values of temperatures measured with the standard thermocouple in the range between 630° and 1,063° C somewhat higher, the maximum difference being about 0.4 degrees [3] near 800° C. The other change was the adoption of a new value (1.438 cm-deg) for the constant c_2 in the radiation formula used to calculate temperatures above the gold point as observed with an optical pyrometer. Also, in the new scale, Planck's radiation formula is specified instead of Wien's for calculating these temperatures. Since Planck's law is consistent with the thermodynamic scale even at high temperatures, this change removed the upper limit to the scale formerly imposed by the use of Wien's law.

The International Temperature Scale is commonly used from its lowest limit, the normal boiling point of oxygen (−182.97° C. or 90.19° K.), to the highest temperatures measured. For temperatures below the oxygen point down to about 10° K, the Bureau maintains an auxiliary scale based on the resistance of capsule-type platinum resistance thermometers, some of which were calibrated using a helium gas thermometer. At the time it was established, this scale was made to agree with the thermodynamic scale as closely as possible. Capsule-type resistance thermometers are calibrated on this scale by the Bureau for laboratories in the United States and in foreign countries.

In connection with maintenance of the International Temperature scale, the Bureau calibrates the three standard instruments—platinum resistance thermometers, platinum-platinum rhodium thermocouples, and optical pyrometers—and certifies them for other laboratories throughout the Nation. These laboratories then use the calibrated instruments as working standards for the calibration of other temperature-measuring instruments. In addition to the working standards, other thermocouples as well as liquid-in-glass thermometers and ribbon-filament lamps are certified by NBS. Standard resistance thermometers and thermocouples are used to determine the temperature of the testing baths and furnaces. Some types of liquid-in-glass thermometers are also calibrated against standard resistance thermometers and are then used as working standards to calibrate other liquid-in-glass thermometers. Approximately 2,500 temperature-measuring instruments—optical pyrometers, ribbon filament lamps, resistance thermometers, thermocouples and liquid-in-glass thermometers—are certified each year for Federal agencies, State and municipal governments, industrial laboratories, and manufacturers. About 48,000 clinical thermometers, a sampling of a much larger number, are also tested each year for the Veterans' Administration, the United States Public Health Service, and the United States Department of Agriculture to insure compliance with specifications. As a result of this standardizing service, practically all measurements of temperatures above the oxygen point in this country are based upon the International Temperature Scale as established and maintained by NBS.

Calibration of a standard platinum resistance thermometer throughout its range is carried out by measuring the resistance of the thermometer at the oxygen point, the ice point, the steam point, and the sulfur point. From these data, constants are calculated for the interpolation equation which gives the resistance of the thermometer at any given temperature in terms of that temperature and the resistance at 0° C. The International Temperature Scale specifies that the platinum in the standard resistance thermometer must be

annealed and must have a purity such that the ratio R_{100}/R_0 is greater than 1.3910.

The standard thermocouple, which must contain platinum of high purity and must satisfy certain specific requirements concerning the electromotive force it develops, is calibrated by measuring its electromotive force when one junction is maintained successively at a temperature between 630.3° C and 630.7° C (as determined by a standard resistance thermometer), at the silver point, and at the gold point. The reference junction of the thermocouple is held at 0° C. These three values of the electromotive force permit the calculation of the three constants in a quadratic equation which relates electromotive force to the corresponding temperature.

Optical pyrometers are used to measure the temperature of incandescent bodies by visual comparison of a portion of the radiation from the hot body with that emanating from an incandescent lamp filament. Calibration of such an instrument involves a determination of the pyrometer lamp current which corresponds to a stated temperature. This is done at NBS by comparison of the pyrometer under study with a previously calibrated standard pyrometer, both instruments being sighted upon a ribbon-filament lamp which serves as a source of constant temperature.

The current-temperature relation for the standard pyrometer is determined by means of Planck's law of radiation. In this process the pyrometer lamp current corresponding to the gold point (1,063° C) is first determined by matching the brightness of a portion of the lamp filament with that of a black body immersed in freezing gold. The calibration is extended above the gold point by using a ribbon-filament lamp and sector disks. The temperature of the ribbon-filament lamp is first adjusted until its brightness, when viewed through a given rotating sector disk, is equal to that of a black body at the gold point. From the known fractional transmission of the sector disk, the brightness temperature of the lamp itself, without the disk, is calculated by means of Planck's law. The sector disk is then removed, and the pyrometer lamp current is increased to obtain a brightness match between the pyrometer lamp and the ribbon filament lamp. When this match is obtained, the pyrometer current corresponds to the calculated temperature. In order to be able to calibrate commercial pyrometers below the gold point, the calibration of the standard pyrometer is extended to lower temperatures by inserting, in turn, various rotating sector disks between the pyrometer and the gold-point black body and observing the pyrometer lamp current required to match the resulting reduced brightness. The corresponding temperatures below the gold point are then calculated from the fraction transmission of the various sectors and Planck's law of radiation. By use of sector disks with different angular openings, the pyrometer lamp currents for various temperatures on either side of the gold point are determined, and a complete calibration is obtained by interpolation.

The most commonly used fixed point in thermometry is the ice point, which is defined as the temperature of equilibrium at a pressure of one atmosphere between ice and air-saturated water. In practice, this definition is difficult to realize exactly because all impurities except air (the composition of which is not specified) must be absent and because complete saturation by air is difficult to attain. Fortunately, the effects of these factors are small except in measurements demanding the highest precision. When the ice point was first defined, the ice bath, made of finely divided pure ice and distilled water exposed to the atmosphere, was assumed to be sufficiently accurate to fix the ice-point temperature within 0.001° C. Later, however, when

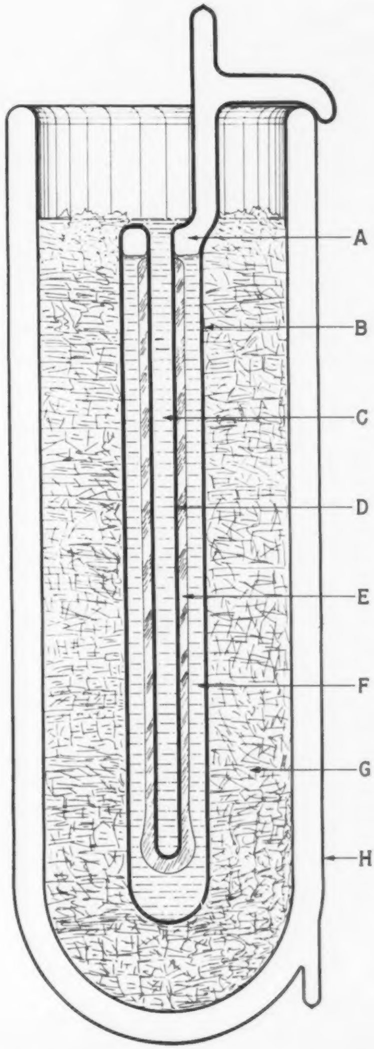

Diagram of the NBS triple-point (*B, D*) in use in an ice bath (*G*) within a Dewar flask (*H*). *A*: water vapor. *C*: thermometer well. *E*: ice mantle. *F*: liquid water.

In testing clinical thermometers, the thermometers are first shaken down in a centrifuge (upper left). They are then heated by immersion in a stirred water bath (lower left), the temperature of which is determined by reading a previously calibrated liquid-in-glass thermometer with the aid of a telescope. The thermometers under test are then removed and their indicated temperatures are determined (above). This procedure is repeated for the other test temperatures.

an accuracy of 0.0001° C. was desired, a more reproducible standard became necessary. The Bureau therefore began investigation of the triple point of water, defined as the temperature at which ice, liquid water, and water vapor are in equilibrium. As a result of this work, a special apparatus known as a triple-point cell [4] was designed for determination of the triple point, and the triple point is now used by the Bureau instead of the ice point for high-precision measurements.

The triple-point cell consists of a cylindrical glass container having a coaxial reentrant well for a thermometer. Very pure air-free water is sealed in the cell, partially filling it. The NBS technique in preparing a cell for use is to freeze a mantle of ice along the thermometer well. This method further purifies the ice adjacent to the thermometer well. A thin layer of ice is then melted to provide the interface between pure water and pure ice, both of which are in contact with the vapor phase. Experiments [5] carried out in 1942 showed that the triple point is very close to 0.0100° C. (0.00997° C.) and that its value as obtained with the triple-point cell is reproducible to within approximately 0.0001° C.

Next in importance to the ice point (or triple point) is the steam point, defined as the temperature of equilibrium between liquid water and its vapor under the standard pressure of 1,013,250 dynes per square centimeter (one atmosphere). The Bureau has conducted considerable research in an effort to make this temperature as reproducible as possible. While the steam point may be determined in a boiling-point apparatus left open to the atmosphere, corrections for atmospheric pressure, which is constantly changing, must be made; and precision is difficult to obtain in this way. The Bureau has therefore developed a controlled pressure system, employing a precision manometer, which has been used for the calibration of standard platinum resistance thermometers. A closed boiler, into which six thermometer wells extend, is connected to the precision manometer by a tube filled with helium. The manometer, located in a temperature-controlled pit, consists of a mercury column with a mercury cell at each end mounted on Hoke gage blocks.

Adjusting the difference in the heights of the two columns of gage blocks establishes the desired pressure, which is transmitted by means of the helium to the closed boiler. In order to determine the heights of the mercury surfaces in the mercury cells, the electrostatic capacity between the mercury surfaces and insulated metal plates above them is made a part of a beat-frequency oscillator circuit. After the manometer is adjusted, a change in pressure is detected by a change in the beat frequency. The pressure can be returned to its original value by the manual adjustment of a small piston in the helium system. With this equipment, a change in pressure equivalent to 0.00001°

C. at the steam point can be detected, and determinations of the steam point made during one day agree within 0.0001° C.

Until recently, standard platinum resistance thermometers were calibrated singly at the sulfur point using apparatus open to atmospheric pressure. However, as a result of NBS research, the use of pressure control has now been extended to measurements of the sulfur point. Previously, variations in atmospheric pressure had been both troublesome and a source of uncertainty in the measurements, and most of the determinations were being made at night because pressure variations are smaller during that period. The new method uses a closed aluminum boiler having wells for 10 thermometers and connected to the precision manometer. This apparatus will make possible a study of the sulfur point as a precise fixed point for thermometry.

The Bureau is now working to obtain the closest possible agreement between the thermodynamic and the International Temperature Scale. To aid in this work, a "noise thermometer" developed at the University of Chicago [6] has been set up at NBS to measure thermodynamic temperatures. The mean-square voltage due to thermal fluctuations in electron density in a resistor—usually referred to as thermal noise—is a function of the thermodynamic temperature of the resistor. By comparing the noise voltages across two resistors at different temperatures, it is possible to determine ratio of the temperatures. The first measurements will be made of the temperature of the gold point, which is important both for the thermocouple and optical-pyrometer ranges of the International Temperature Scale.

Fixed Points* [a] *of the International Temperature Scale of 1948

Fixed point	Temperature
	° C
Oxygen point	−182.97
Temperature of equilibrium between liquid oxygen and its vapor.	
Ice Point (fundamental fixed point)	0.00
Temperature of equilibrium between ice and air-saturated water.	
Steam point (fundamental fixed point)	100.00
Temperature of equilibrium between liquid water and its vapor.	
Sulfur point	444.60
Temperature of equilibrium between liquid sulfur and its vapor.	
Silver point	960.80
Temperature of equilibrium between solid and liquid silver.	
Gold point	1,063.00
Temperature of equilibrium between solid and liquid gold.	

[a] Under the standard pressure of 1,013,250 dynse/cm^2.

Closely related to the work on the temperature scale is the development of improved temperature-measuring instruments. Thus, in connection with the maintenance and improvement of the International Temperature Scale in the temperature range from 630.5° to 1063° C., investigations [7] have been made to determine the effect of annealing on the electromotive force of standard platinum versus platinum-rhodium thermocouples. This study included a determination of the effects of different annealing temperatures, cooling rates, and atmospheres in which the thermocouples were cooled. It was found that some of the electrical properties depend upon the rate of cooling and that not only chemical purity but also mechanical strains must be controlled for maximum uniformity.

A search is now under way for a better material to substitute for the alumel wire of a chromel-alumel thermocouple. When used under conditions encountered in exhaust gases in aircraft engines, the alumel becomes brittle and fails after a relatively short period of operation. Available substitutes for alumel which appear likely to withstand the vibration and corrosive atmosphere are being investigated to determine their performance under these extreme conditions.

Future plans call for the construction of comparator furnaces to study the performance of standard platinum resistance thermometers and thermocouples between calibration points. As the International Temperature Scale is based on a relatively small number of fixed points, it is necessary to investigate the standard temperature-measuring instruments between these points and to evaluate the precision with which they can be used over the intervening ranges. In this way the Bureau hopes to ensure that standard calibrated instruments of a given type indicate the same temperature at any temperature within their range.

Investigative work is also carried on in connection with the testing of clinical thermometers. Recently, in order to develop an improved specification for clinical thermometers, a study [8] was made of the change with time in the calibration of a group of thermometers which had been given a special stabilizing heat treatment by the manufacturer. Previous specifications had required that clinical thermometers be aged by three months' storage before calibration so that the volumes of the bulbs would have time to become constant. The results of the Bureau's study showed that the "artificial aging," which requires less than a week, is fully equivalent to the earlier method.

A significant advance in the measurement of extremely high temperatures was the recent development of an iridium-iridium-rhodium thermocouple by the NBS combustion section. Because of the high temperatures up to about 3,800° F—which prevail in the primary burning zones of turbojet and ramjet combustion chambers, conventional temperature-sensing instruments are not suitable for use in these applications. In preliminary studies, the new thermocouple has been found to withstand both the thermal and mechanical stresses incident to combustion-chamber operation, and heat-resistant supporting tubes and insulators are now being developed so that the device can be used in flight.

At very low temperatures (below 50° K.) NBS research on thermometry has two major objectives: First, the determination of thermodynamic temperatures by means of a gas thermometer and, second, the development of convenient, sensitive, and reproducible secondary thermometers which can be calibrated by means of the gas thermometer. Since the development

of a highly accurate gas thermometer for this purpose requires painstaking and time-consuming precision, the work on the secondary thermometer is being pursued concurrently. Resistance thermometers constructed of the semiconducting elements, silicon and germanium, have proved to be extremely sensitive; in some cases the resistance changes more than 50 percent per degree. While satisfactory reproducibility still remains a problem, results of initial tests have been quite promising.

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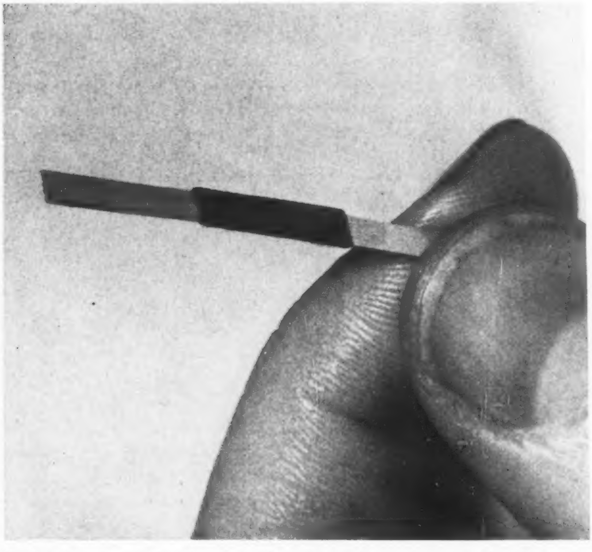
NBS Precured Tape Resistor

THE ADHESIVE-TAPE resistor developed by the Bureau has aroused wide interest since its announcement in 1951.[1] In the NBS tape-resistor system, designed primarily for electronic printed-circuit applications, small pieces of self-adhesive resistance-coated tape are simply pressed into place against metallic terminals at the proper points in the circuit. The resistor was developed as part of a program of miniaturization of airborne equipment sponsored by the Navy Bureau of Aeronautics. Despite its advantages, the method has been limited in some applications by the necessity for baking the supporting base material to cure the resistors after they have been pressed in place.

A new precured wire-lead version of the tape resistor, now being made at NBS, obviates the need for heat-curing after placement in the circuit. The new resistors are made by pressing uncured resistor tape against both sides of suitable wire or metal-ribbon leads; the leads are thus sandwiched between two pieces of resistor tape. These units are then given the usual heat cure, which bonds the resistor tape to the

[1] A high-temperature adhesive tape resistor, *NBS Tech. News Bull.* **35**, *100 (July 1951). Described in detail in* An adhesive tape resistor system, *NBS Circular 530, Government Printing Office, 30¢.*



Left: **the recently developed precured version of the Bureau's tape resistor can be soldered or spot-welded into the circuit. The original version of the NBS tape resistor is self-adhesive, but must be heat-cured by baking the chassis after all resistors have been pressed in place. The precured resistor is made by sandwiching suitable metal leads between two uncured resistors and then heat-curing, which bonds the resistor to the leads. Over-all length is about 1½ inches.** ***Right:*** **soldering one of the precured NBS tape resistors into place. Because no subsequent heat-curing is needed, this version of the tape resistor can be used with chassis that would not withstand curing temperatures (about 300° C.).**

leads and results in resistors that may be soldered or spot-welded into the circuit.

With the new precured variation in addition to the basic press-on form, the range of possible applications of the NBS tape resistor is greatly extended. Characteristic advantages of the NBS tape resistor—compactness, stability, and high-temperature operation—are largely retained in the precured wire-lead design. Furthermore, the new resistor might well prove more economical to manufacture in quantity than other types having less desirable characteristics.

The basic NBS tape resistor is made by coating asbestos-paper tape with a mixture of carbon black or graphite, silicone resin, and solvent. Resistor dimensions are standardized at one-half inch long and about one-eighth inch wide; a variety of coating formulations have been developed to give a wide range of resistor values.

Leads for the precured tape resistor are now being made from ribbon of thin silver or silver-plated copper at NBS. Leads extending one-half inch beyond the resistor proper are used, bringing the over-all length to $1\frac{1}{2}$ inches. Thickness is held to about 0.012 to 0.015 inch.

Preliminary tests indicate that the precured NBS tape resistor, when supported in air by its leads alone, will not provide the full dissipation of 0.25 watt at 200° C for which the basic resistor was designed. Further test work is now in progress, and a suitable derating curve will be worked out.

New NBS Director Appointed

DR. ALLEN V. ASTIN has been appointed* Director of the National Bureau of Standards. Formerly Associate Director of the Bureau, Dr. Astin has been Acting Director since October 1951. Dr. Astin has also been appointed a member of the National Advisory Committee for Aeronautics.

Dr. Astin has been a member of the Bureau's staff since 1932. Until 1940 he was principally concerned with dielectrics and electronics. His contributions include development of improved methods for precise measurement of dielectric constants and power factors of dielectric materials and studies of the nature of energy losses in air capacitors. He did pioneering work in the development of radio telemetering techniques and instruments and applied this work to studies of cosmic rays and of meteorological problems in the earth's upper atmosphere.

In 1940 Dr. Astin was one of the Bureau scientists doing pioneering work in proximity fuze research and development for bombs and rockets. He became chief of the Optical Fuze Section in 1943, assistant chief of the Ordnance Development Division in November 1943, and chief of the Division in July 1948. He played a major part in the development and evaluation of bar-type proximity bomb fuzes and in their introduction to service during the war. During the fall and winter of 1944–45 he served in Europe as representative of the Bureau and consultant for the Ordnance Accessories Division of the National Defense Research Committee, concentrating on proximity fuze problems. He edited the terminal three-volume Technical Report of the Ordnance Accessories Division (Division 4).

As chief of the Ordnance Division from 1948 to 1950, he supervised the Ordnance Laboratory, the Guided Missile Laboratories, and the Electronics and Tube Laboratories. When Dr. Astin was appointed Associate Director in May 1950, he assumed responsibility for the work of the Ordnance Development, Missile Development, Electricity, and Electronics Divisions as well as the Office of Basic Instrumentation.

Dr. Astin was born in Salt Lake City, Utah, on June 12, 1904. He received the B. S. degree in physics from the University of Utah in 1925. While working toward his advanced degrees at New York University from 1925 to 1928, he was a graduate assistant and instructor in physics. From N. Y. U. he obtained the M. S. and Ph.D. degrees in physics in 1926 and 1928 respectively. From 1928 to 1930 he held a National Research Council Fellowship at Johns Hopkins University, doing basic research on measurement techniques relating to dielectric materials. Between 1930 and 1932, he was a Research Associate in a program sponsored at the Bureau by the National Research Council and the Utilities Research Commission, Inc.

Honors and awards he has received include the following: National Research Council Fellow in Physics, 1928–1930; Navy Ordnance Award for Exceptional

Dr. Allen V. Astin

Service, 1945; Army Ordnance Award for Outstanding Service, 1946; Presidential Certificate of Merit, 1948; His Majesty's Medal for Service in the Cause of Freedom (from Great Britain), 1947; Gold Medal Exceptional Service Award, Department of Commerce, 1952.

Dr. Astin is a Fellow of the American Physical Society and the American Association for the Advancement of Science, and a member of the Institute of Radio Engineers, Sigma Xi, American Ordnance Association, Washington Philosophical Society, Washington Academy of Sciences, and the Cosmos Club.

*On May 20, 1952, President Truman nominated Dr. Astin as Director of the Bureau; the Senate confirmed the nomination on May 28, 1952; he was sworn in as Director by Secretary of Commerce Charles Sawyer on June 12, 1952.

Proceedings of the Second Technical Session on Bone Char, 1951

THE PROCEEDINGS of the Second Technical Session on Bone Char, held at the Bureau in May 1951, are now available. The technical session, which was sponsored by NBS and those industries supporting the bone-char research, offered representatives of the sponsoring sugar refineries and bone-char manufacturers an opportunity to present current practical problems and to learn the present status of the research program of the Bone Char Research Project, Inc. Four sessions covered the general subjects of test procedures, defecation preliminary to char filtration, filtration operations, and bone-char kilns.

The test procedures section, available separately, is expected to form the nucleus of standard methods for testing bone char. Until now lack of uniformity in testing procedures has handicapped the refining industry.

Proceedings of the Second Technical Session on Bone Char, 1951, 436 large pages, figures, charts, and half tones, $4 prepaid. Reprints of section on "Test Procedures" (pp. 15–134), $2 prepaid. (Order from J. M. Brown, Secretary-Treasurer, Bone Char Research Project, Inc., care of Revere Sugar Refinery, 333 Medford Street, Charlestown 29, Mass.)

Publications of the National Bureau of Standards

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Journal of Research of the National Bureau of Standards, volume **48**, number 6, June 1952 (RP2329 to RP2335, incl.)

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RP2322. Influence of temperature and moisture on the electrical properties of leather. Charles E. Weir.

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RP2325. Wavelengths of rotational lines in the water-vapor bands of 0.93 and 1.13 microns. C. C. Kiess.

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